# Silacyclopentadiene Complexes of Molybdenum, Chromium, Iron, Ruthenium, Cobalt, and Rhodium

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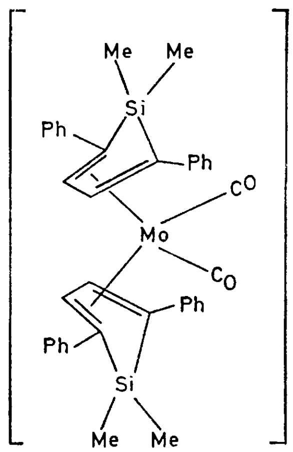
The synthesis of a series of transition-metal derivatives of various substituted silacyclopentadienes is described. A number of stoicheiometries and modes of bonding of the silacyclopentadiene molecule are reported. Molybdenum hexacarbonyl forms the bis(diene) complex $[Mo(C_4Ph_2H_2SiMe_2)_2(CO)_2]$, but the same ligand forms only an arene complex with chromium hexacarbonyl, $[Cr(C_4Ph_2H_2SiMe_2)(CO)_3]$. Both $[Co_2(C_4Ph_2H_2SiMe_2)(CO)_6]$ and $[Co_2(C_4Ph_2H_2SiMe_2)_2(CO)_4]$ have been observed but only the latter could be isolated. Rhodium complexes formulated as $[\{Rh(C_4Ph_2H_2SiMe_2)Cl\}_2]$ and $[\{Rh(CO)_2Cl\}_2(C_4Ph_2H_2SiMe_2)]$ are reported and the latter is believed to involve a bridging silacyclopentadiene group. 1-Hydrido-1-methyl-2,3,4,5-tetraphenyl-1-silacyclopentadiene forms exclusively the *endo*-H form of $[Fe(C_4Ph_4SiMeH)(CO)_3]$ on reaction with $[Fe_3(CO)_{12}]$, but the same silacyclopentadiene and $[Ru_3(CO)_{12}]$ form $[\{Ru(C_4Ph_4SiMe)(CO)_4\}_2]$ containing a Si–Ru–Ru–Si sequence. $\eta^4$-Diene-bonded silacyclopentadiene complexes of ruthenium, $[Ru(C_4Ph_2H_2SiMe_2)(CO)_3]$ and $[Ru(C_4Ph_4SiPhCl)(CO)_3]$, are also reported. Structural and spectroscopic characteristics of these complexes are reported and compared and contrasted with related species.

IN addition to their intrinsic interest as cyclic diene derivatives, the transition-metal complexes of silacyclopentadiene have a particular importance as potential sources of $\eta$-silacyclopentadienylmetal complexes. To date, neither the silacyclopentadienyl anion nor its $\eta^5$-metal derivatives have been isolated, despite their observation as transient species, especially in mass spectra.[1,2] The metal complexes of silacyclopentadienes have been restricted to those of iron [1,3-7] and cobalt,[2,8] and a preliminary report of our own work on ruthenium.[9] It was the aim of the present study to extend the range of metals complexing with silacyclopentadienes.

## RESULTS AND DISCUSSION

1,1-Dimethyl-2,5-diphenyl-1-silacyclopentadiene underwent markedly different reactions with molybdenum and chromium hexacarbonyls. Prolonged (40 h) reaction of the ligand with $[Mo(CO)_6]$ produced a red crystalline product, for which from analytical data, infrared and n.m.r. spectra, and mass spectrometry we propose the formulation dicarbonylbis(1,1-dimethyl-2,5-diphenyl-1-silacyclopentadiene)molybdenum, (1). This complex is analogous to other [10,11] bis(diene) complexes of molybdenum carbonyl such as bis(buta-1,3-diene)dicarbonylmolybdenum and dicarbonylbis(tetraphenylcyclopentadienone)molybdenum.

In contrast, reaction of the ligand with $[Cr(CO)_6]$ produced tricarbonyl(1,1-dimethyl-5-phenyl-1-silacyclopentadien-2-ylbenzene)chromium, (2), in which we infer that the chromium tricarbonyl moiety is bonded to an arene ring. This preference of chromium for bonding to arenes rather than dienes when the alternatives are available is not uncommon,[12] as exemplified in tricarbon-



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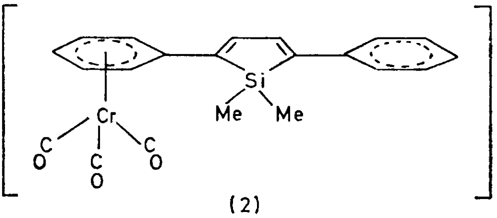

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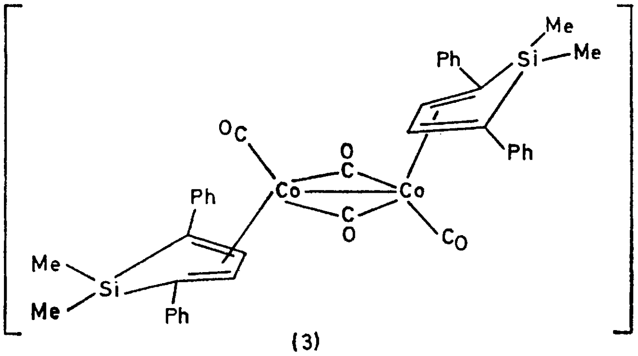
yl(1,4-diphenylbutadiene)chromium. There appeared to be little reaction between the ligand and [W(CO)$_6$], and we were unable to isolate any crystalline product.

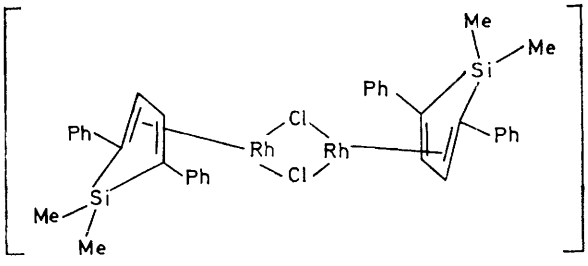
Dicobalt octacarbonyl and the ligand underwent reaction at room temperature with evolution of carbon monoxide. When there was no further change in the metal-carbonyl region of the i.r. spectrum the presence of [Co$_2$(C$_4$Ph$_2$H$_2$SiMe$_2$)(CO)$_6$] and [Co$_2$(C$_4$Ph$_2$H$_2$SiMe$_2$)$_2$-(CO)$_4$] was inferred from the spectra, especially by comparison [13,14] of the reactions of other dienes with [Co$_2$-(CO)$_8$]. During work up of the products, however, we were only able to isolate bis[dicarbonyl(1,1-dimethyl-2,5-diphenyl-1-silacyclopentadiene)cobalt], (3). The difficulty in obtaining the monodiene complexes of cobalt carbonyl has been noted [13] previously, and could be due to a tendency for them to disproportionate to the bis-(diene) complex and cobalt carbonyls. The complex [{Co(C$_4$Ph$_2$H$_2$SiMe$_2$)(CO)$_2$}$_2$] was isolated as an air-stable red crystalline solid. The metal-carbonyl region of the i.r. spectrum was independent of solvent and showed one terminal and one bridging carbonyl band. This strongly suggests the centrosymmetric species (3). From the i.r. spectra there is no evidence of the presence of isomers, and the $^1$H n.m.r. spectrum is consistent with the presence of one isomer, with both the silacyclopentadiene rings in identical environments.



(3)

Bis(dicarbonylchlororhodium) and the ligand gave two products which were easily separated, due to very different solubilities. The minority product, which from analytical data, n.m.r. spectra, and the absence of CO absorption in the i.r. spectrum was formulated as [{Rh(C$_4$Ph$_2$H$_2$SiMe$_2$)Cl}$_2$], is closely analogous to other [{Rh(diene)Cl}$_2$] complexes and is believed to have the structure (4). In the absence of diffraction evidence, however, we are unable to say whether the central Rh$_2$Cl$_2$ is planar as in di-μ-chloro-bis(cyclo-octa-1,5-dienerhodium) [15] or bent as in [{Rh(CO)$_2$Cl}$_2$].[16] The major product of this reaction, however, appears to be an ' addition ' complex of the silacyclopentadiene and rhodium carbonyl chloride without the loss of CO. From the presence of the CO stretching frequencies and the n.m.r. spectrum for this complex we propose structure (5) in which the rhodium atoms are bridged both by chlorine and a silacyclopentadiene group. Such bonding would enable the formally 16-electron shell of each rhodium in the [{Rh-(CO)$_2$Cl}$_2$] molecule to attain an 18-electron rare-gas configuration. Some other diene complexes of [{Rh-(CO)$_2$Cl}$_2$] have been noted previously,[17] but unfortunately there are no structural data available.

The preparation, reactions, and stereochemistry about silicon of a number of tricarbonyliron derivatives of silacyclopentadienes have been reported.[1,3-7] We have extended these studies in particular to the reaction of a 1-hydrido-1-silacyclopentadiene with iron carbonyls. This was of particular interest due to the possible formation of silicon–iron bonds [18] by loss of hydrogen, in addition or alternatively to dieneiron complex formation.



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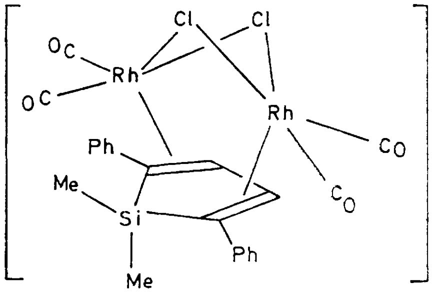

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C$_4$Ph$_2$H$_2$SiMe$_2$ + [{Rh(CO)$_2$Cl}$_2$] → [{Rh(C$_4$Ph$_2$H$_2$SiMe$_2$)Cl}$_2$] (10%)

C$_4$Ph$_2$H$_2$SiMe$_2$ + [{Rh(CO)$_2$Cl}$_2$] → [{Rh(CO)$_2$Cl}$_2$(C$_4$Ph$_2$H$_2$SiMe$_2$)] (57%)

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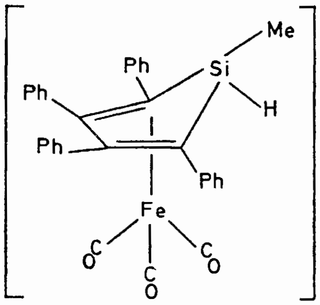
1-Hydrido-1-methyl-2,3,4,5-tetraphenyl-1-silacyclopentadiene underwent reaction with tri-iron dodecacarbonyl to form [Fe($C_4Ph_4SiMeH$)$(CO)_3$] (6). The $^1H$ n.m.r. spectrum of (6) is consistent with an *exo*-methyl, *endo*-hydrogen structure, and no trace of the alternative isomer was noted. Interestingly, both isomers have recently been reported[7] from the reduction of tricarbonyl-(*exo*-1-methoxy-*endo*-1-methyl-2,3,4,5-tetraphenyl-1-silacyclopentadiene)iron, depending on the reaction conditions. In contrast, the reaction of $C_4Ph_4SiMeH$ with triruthenium dodecacarbonyl yields the product formulated as [{Ru($C_4Ph_4SiMe$)$(CO)_4$}$_2$] (8) from analytical data, molecular-weight measurements, and i.r. and n.m.r. spectra. This is believed to be formed by the sequence illustrated above. The proposed oxidative addition of the hydridosilane is similar to those in which such products have been isolated,[19] and is analogous to the first step in catalysed hydrosilylation of olefins. The Si–Ru–Ru–Si sequence in (8) is already well characterised[20] in a range of complexes of the trialkylsilanes.



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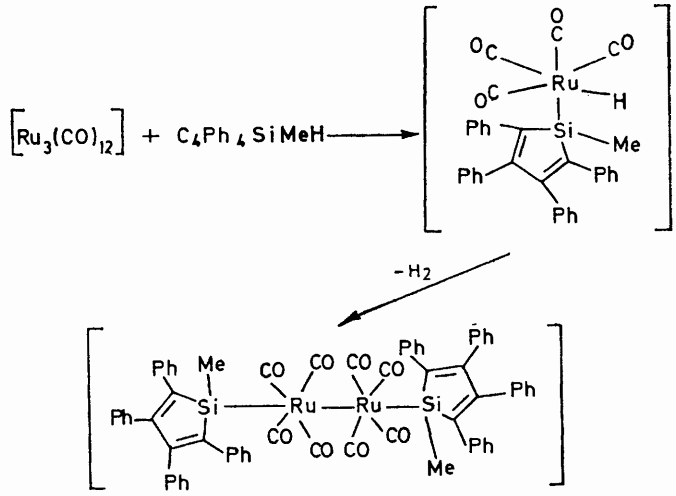

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The alternative $\eta^4$-diene bonding to ruthenium carbonyl is obtained for silacyclopentadienes other than the 1-hydrido-species. Thus 1,1-dimethyl-2,5-diphenyl- and 1-chloro-1,2,3,4,5-pentaphenyl-1-silacyclopentadiene underwent reaction with [$Ru_3(CO)_{12}$] to produce the corresponding tricarbonylruthenium complexes (9) and (10), as evidenced from analytical data and the characterisations in Tables 1 and 2 respectively.

The molecular structure of $C_4Ph_2H_2SiMe_2$ and its tricarbonylruthenium complex have been reported from *X*-ray diffraction measurements.[9] The silacyclopentadiene itself possesses $C_s$ symmetry, and the bond lengths are consistent with completely localised double bonds. The silicon atom is displaced from the four-carbon-atom plane by only 0.08 Å, giving a dihedral angle of only 3.7°. Whilst the approximately $C_s$ symmetry is retained in the tricarbonylruthenium complex, the ruthenium atom is $\eta^4$-bonded to the butadiene unit of the silacyclopentadiene ring, with considerable perturbation of the C–C bond lengths suggesting delocalisation, as in (11). Further, the $SiC^1C^4$ (butadiene) dihedral angle has increased to 32°, so that the silicon atom lies 0.72 Å from the butadiene plane. The Ru–Si distance of 2.99 Å excludes any possibility of normal bonding between these two atoms since the Ru–Si covalent bond length is known[21] to be 2.43 Å. Such non-bonding between the ring heteroatom in other heterocyclopentadiene metal complexes has been noted[22] to result in similar dihedral angles of *ca.* 30°. On the basis of this structural determination of the tricarbonylruthenium complex, we believe it is likely that the other silacyclopentadiene metal carbonyl complexes reported by us and others will have similar bent rings.

We have observed the formation of two 1-hydroxy-1-silacyclopentadiene complexes. A minority product

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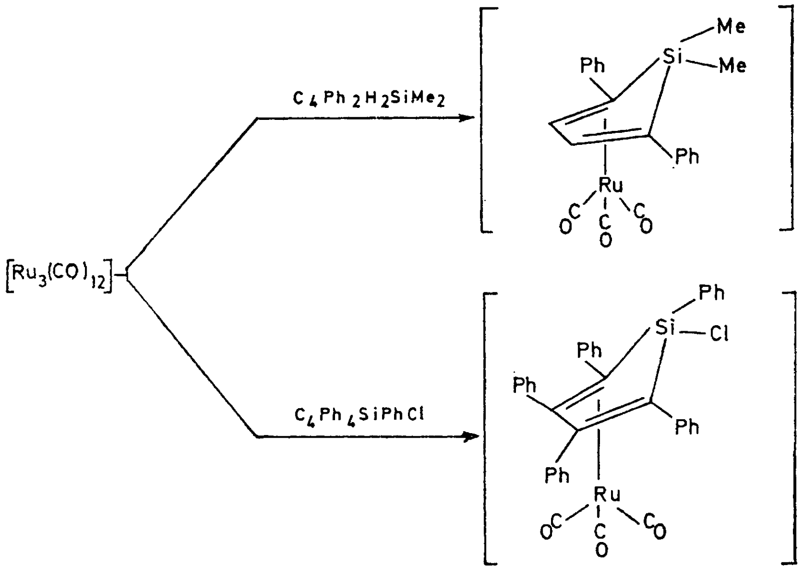
from the reaction of $C_4Ph_4SiMeH$ and $[Ru_3(CO)_{12}]$ was characterised by mass spectrometry only as tricarbonyl(1-hydroxy-1-methyl-2,3,4,5-tetraphenyl-1-silacyclopentadiene)ruthenium. It is possible that a small quantity of the original ligand underwent accidental hydrolysis, thus preventing it undergoing the proposed oxidative-addition reaction to form (7), and hence alternatively forming a diene type of complex. Similarly the interaction of $C_4Ph_4SiPhCl$ with $[Ru_3(CO)_{12}]$ whilst producing (8) as the major product, also produced a small amount of tricarbonyl(1-hydroxy-1,2,3,4,5-pentaphenyl-1-silacyclopentadiene)ruthenium (characterised by mass



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(10)

TABLE 1

Metal π-complexes of 1,1-dimethyl-2,5-diphenyl-1-silacyclopentadiene

| | | $^1$H N.m.r. (τ) [b] | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Compound | Carbonyl i.r. spectra (cm$^{-1}$) [a] | C=CH and C=CPh | Ph | π-$C_6H_5$ | C=CH | Me | Me(*endo*) | Me(*exo*) |
| $C_4Ph_2H_2SiMe_2$ | | 2.50—2.79(12) | | | | 9.47(6) | | |
| $[Mo(C_4Ph_2H_2SiMe_2)_2(CO)_2]$ | 1 984(10),<br>1 942 (br, 6.1) | | 2.69—2.78(20) | | 4.93(4) | | 9.13(6) | 10.29(6) |
| $[Cr(C_4Ph_2H_2SiMe_2)(CO)_3]$ | 1 977(10),<br>1 915(6.3),<br>1 912(6.3) | 2.56—2.78(7) | | 4.42—4.64(5) | | 9.42(6) | | |
| $[Ru(C_4Ph_2H_2SiMe_2)(CO)_3]$ | 2 062(10),<br>1 998 (br, 8.6) | | 2.80—2.84(10) | | 3.98(2) | | 9.04(3) | 9.94(3) |
| $[\{Co(C_4Ph_2H_2SiMe_2)(CO)_2\}_2]$ | 2 020(10),<br>1 997(1.5),<br>1 842(4.5) | | 2.63(20) | | 3.92(4) | | 8.92(6) | 10.02(6) |
| $[\{Rh(C_4Ph_2H_2SiMe_2)Cl\}_2]$ | | | 2.56—2.73(20) | | 4.16(4) | | 8.57(6) | 9.82(6) |
| $[\{Rh(CO)_2Cl\}_2(C_4Ph_2H_2SiMe_2)]$ | 2 086(9.5),<br>2 060(7.5),<br>2 036(1.3),<br>2 016(10) | | 2.28—2.64(10) | | 3.63(2) | | 8.84(3) | 9.90(3) |

[a] In cyclohexane solution; relative peak heights are given in parentheses. [b] In $CDCl_3$, with reference to $SiMe_4$. Relative intensities are given in parentheses.

TABLE 2

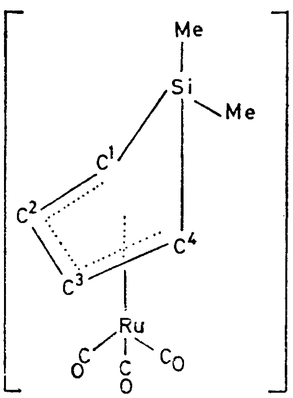
Miscellaneous silacyclopentadiene complexes

| | | $^1$H N.m.r. spectra (τ) [b] | | |
|---|---|---|---|---|
| Compound | Carbonyl i.r. spectra (cm$^{-1}$) [a] | Ph | Si–H | Me |
| $C_4Ph_4SiMeH$ | | 2.64—3.09(20) | 4.91 (q, 1) | 9.43 (d, 3) |
| $[Fe(C_4Ph_4SiMeH)(CO)_3]$ | 2 046(10), 1 988(5.3), 1 976(6) | 2.73—2.94(20) | 4.30 (q, 1) | 9.62 (d, 3) |
| $[\{Ru(C_4Ph_4SiMe)(CO)_4\}_2]$ | 2 055(1.8), 2 027 (sh, 7.5), 2 022(10) [c] | 2.88—3.18(40) | | 8.85(6) |
| $[Ru(C_4Ph_4SiPhCl)(CO)_3]$ | 2 070(10), 2 016(9.2), 1 997(8.9) | | | |

[a] In cyclohexane; relative peak heights are given in parentheses. [b] In $CDCl_3$, with reference to $SiMe_4$. Relative intensities are given in parentheses. [c] In $CS_2$.

spectrum only), presumably by accidental hydrolysis of the silicon-chlorine bond in a small amount of the 1-chloro-1-silacyclopentadiene.

The mass spectra of the various silacyclopentadiene complexes reported herein have a number of interesting features, especially ions corresponding to η-silacyclopentadienyls. Thus, for example, the mass spectrum of $[Ru(C_4Ph_2H_2SiMe_2)(CO)_3]$ contained, in addition to the parent molecular ion and fragments corresponding to successive loss of the three carbonyl groups, a fragmentation sequence in which one of the methyl groups was lost. In the corresponding cyclopentadieneiron complexes this is attributed to the preferential loss of the *exo* substituent.[23]



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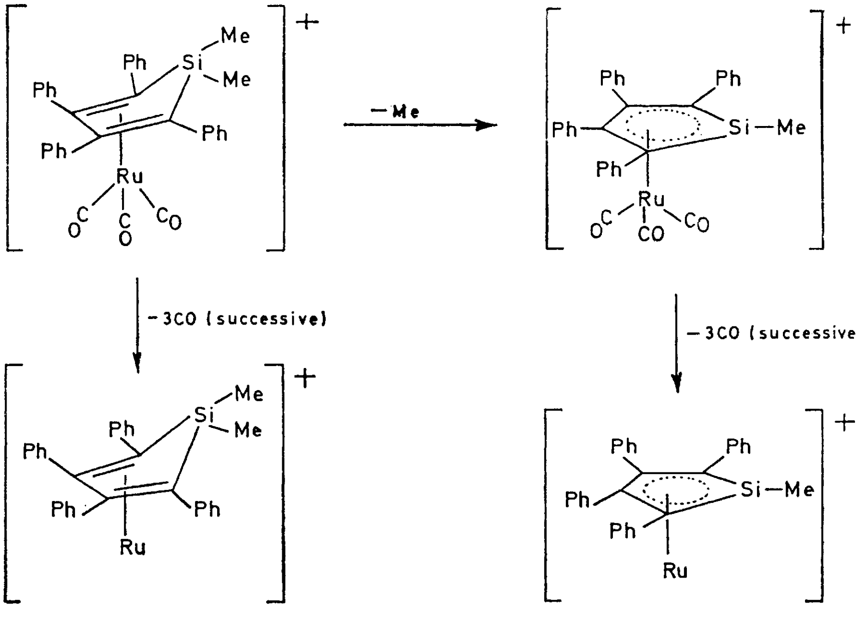
$C^1-C^2 = 1.45$ Å
$C^2-C^3 = 1.41$ Å
$C^3-C^4 = 1.45$ Å



## EXPERIMENTAL

All the reactions were routinely carried out under an atmosphere of nitrogen using anhydrous solvents. Infrared spectra were recorded on a Perkin-Elmer model 257 spectrometer, n.m.r. spectra on a Jeol 100 MHz instrument, and mass spectra on A.E.I. MS9 and Hitachi–Perkin-Elmer RMU6 spectrometers. Microanalyses were by the Department of Chemistry at Exeter and Pascher Microanalytical Laboratories, Bonn, West Germany.

Silacyclopentadienes were prepared by literature methods.[18, 24]

*Interactions of* 1,1-*Dimethyl*-2,5-*diphenyl*-1-*silacyclopentadiene*.—*With molybdenum hexacarbonyl.* The silacyclopentadiene (0.16 g, 0.6 mmol) was heated with $[Mo(CO)_6]$ (0.32 g, 1.2 mmol) in toluene (10 $cm^3$) under reflux (40 h). Solvent was removed (20 °C, 0.01 mmHg)* and the oily residue was extracted with hexane to give an orange solution, and a small amount of yellow precipitate identified as tricarbonyl(toluene)molybdenum. The hexane solution was chromatographed on Florisil. Elution with toluene–hexane (2 : 1) yielded dicarbonylbis(1,1-dimethyl-2,5-diphenyl-1-silacyclopentadiene)molybdenum, (1) (0.094 g, 44%), which recrystallised from hexane as deep orange crystals, m.p. 115—116 °C (Found: C, 67.5; H, 5.73. $C_{38}H_{36}MoO_2Si_2$ requires C, 67.4; H, 5.36%). Mass spectrum: $M^+$, 678 (15); $[M - Me]^+$, 663 (0.6); $[M - CO]^+$, 650 (2.3); $[M - (CO)_2]^+$, 622 (100).

*With chromium hexacarbonyl.* This reaction was carried out and worked-up essentially as described above. The only product isolated was tricarbonyl(1,1-dimethyl-5-phenyl-1-silacyclopentadien-2-ylbenzene)chromium, (2) (0.123 g, 58%) as yellow-orange crystals, m.p. 159—161 °C (decomp.) (Found: C, 64.0; H, 4.60. $C_{21}H_{18}CrO_3Si$ requires C, 63.4, H, 4.55%). Mass spectrum: $M^+$, 398 (15); $[M - CO]^+$, 370 (0.2); $[M - (CO)_2]^+$, 342 (8.8); $[M - (CO)_3]^+$, 314 (100).

*With dicobalt octacarbonyl.* The silacyclopentadiene (0.18 g, 0.7 mmol) was stirred with the carbonyl (0.12 g, 0.35 mmol) in hexane (3 h) at room temperature. The reaction was continuously monitored by the i.r. spectrum in the

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* 1 mmHg ≈ 13.6 × 9.8 Pa.

[23] J. Müller, G. E. Herberich, and H. Müller, *J. Organometallic Chem.*, 1973, **55**, 165.

[24] W. H. Atwell, D. R. Weyenberg, and H. Gilman, *J. Org. Chem.*, 1967, **32**, 885.

metal-carbonyl region. After 3 h the spectrum had bands at 2 074vs, 2 025br,s, 2 016s, 2 010s(sh), 1 866w, 1 854s, and 1 844m(sh) cm$^{-1}$, and appeared to have reached equilibrium. From a study of comparable spectra [13,14] of dienecobalt carbonyl complexes it appears that [$Co_2(CO)_6(C_4Ph_2H_2SiMe_2)$] and [$Co_2(CO)_4(C_4Ph_2H_2SiMe_2)$] were both present in solution. The solution was worked-up by fractional crystallisation on cooling the cyclohexane solution. Despite the apparent presence of two products in solution, only red needles of bis[dicarbonyl(1,1-dimethyl-2,5-diphenyl-1-silacyclopentadiene)cobalt], (3) (0.27 g, 52%), m.p. 129 °C (decomp.) (Found: C, 63.3; H, 4.80. $C_{40}H_{36}Co_2O_4Si$ requires C, 63.6; H, 4.80%), were obtained as product.

*With bis(dicarbonylchlororhodium).* The silacyclopentadiene (0.22 g, 0.84 mmol) and the rhodium dimer (0.18 g, 0.46 mmol) were heated under reflux in hexane (1 h). On cooling a red precipitate was formed, which after recrystallisation from dichloromethane–hexane gave bis[chloro(1,1-dimethyl-2,5-diphenyl-1-silacyclopentadiene)rhodium], (4) (0.035 g, 10%), m.p. 238—239 °C (decomp.) (Found: C, 52.9; H, 4.55. $C_{36}H_{36}Cl_2Rh_2Si_2$ requires C, 53.9; H, 4.55%), as red crystals. Fractional crystallisation of the supernatant hexane solution from the original reaction produced red crystals of [$\{Rh(CO)Cl\}_2(C_4Ph_2H_2SiMe_2)$] (5) (0.17 g, 57%), m.p. 107—110 °C (decomp.) (Found: C, 40.3; H, 3.10. $C_{22}H_{18}Cl_2O_4Rh_2Si$ requires C, 40.5; H, 2.79%).

*Interactions of* 1-*Hydrido*-1-*methyl*-2,3,4,5-*tetraphenyl*-1-*silacyclopentadiene.*—*With tri-iron dodecacarbonyl.* The silacyclopentadiene (0.4 g, 1 mmol) and the carbonyl were heated (12 h) under reflux in toluene. The originally dark green solution became deep orange. Solvent was removed (20 °C, 0.01 mmHg) and the remaining orange oil was extracted with hexane and chromatographed on Florisil to yield yellow-orange crystals of tricarbonyl(1-hydrido-1-methyl-2,3,4,5-tetraphenyl-1-silacyclopentadiene)iron, (6) (0.19 g, 35%), m.p. 170 °C (decomp.) (Found: C, 71.7, H, 4.70. $C_{32}H_{24}FeO_3Si$ requires C, 71.1, H, 4.50%). Mass spectrum: $M^+$, 540 (4); $[M - Me]^+$, 525 (0.5); $[M - CO]^+$, 512 (20); $[M - (CO)_2]^+$, 484 (1.5); $[M - Me - (CO)_2]^+$, 469 (18); $[M - (CO)_3]^+$ 456 (100); $[M - Me - (CO)_3]^+$, 441 (1).

*With triruthenium dodecacarbonyl.* The silacyclopentadiene (0.53 g, 1.3 mmol) and the carbonyl (0.35 g, 0.55 mmol) were heated in toluene under reflux for 12 h. Removal of solvent (20 °C, 0.01 mmHg) and addition of hexane produced a yellow precipitate and a red solution. The yellow solid was recrystallised from hexane–dichloromethane, to give bis[tetracarbonyl(1-methyl-2,3,4,5-tetraphenyl-1-silacyclopentadiene)ruthenium], (8) (0.86 g, 53%), m.p. 200—205 °C (decomp.) [Found: C, 64.7; H, 4.05; Ru, 16.45; Si, 4.55. $C_{66}H_{46}O_8Ru_2Si_2$ requires C, 64.7; H, 3.80; Ru, 16.5; Si, 4.60%). $M$ (Osmometrically in benzene) 1329 (required 1225)]. The red cyclohexane solution from the original extraction was chromatographed on Florisil and fractions eluted included [$Ru_3(CO)_{12}$], [$Ru_4(CO)_{12}H_4$], and [$Ru_4(CO)_{13}H_2$]. Hexane–toluene (4 : 1) eluted tricarbonyl(1-hydroxy-1-methyl-2,3,4,5-tetraphenyl-1-silacyclopentadiene)ruthenium, m.p. 163—168 °C (decomp.). Mass spectrum: $M^+$, 602 (4.6); $[M - CO]^+$, 574 (9.2); $[M - (CO)_2]^+$, 546 (15.5); $[M - (CO)_3]^+$, 518 (46); $[C_4Ph_4SiMe(OH)]^+$, 416 (100).

*Interaction of* 1,1-*Dimethyl*-2,5-*diphenyl*-1-*silacyclopentadiene and Triruthenium Dodecacarbonyl.*—The silacyclopentadiene (0.74 g, 3 mmol) and [$Ru_3(CO)_{12}$] (0.65 g, 1 mmol) were heated under reflux in toluene (7 h). Solvent was removed (20 °C, 0.01 mmHg) and the hexane extract of the residue was chromatographed on neutral alumina. Elution with toluene afforded tricarbonyl(1,1-dimethyl-2,5-diphenyl-1-silacyclopentadiene)ruthenium, (9) (0.72 g, 53%), m.p. 122 °C, (Found: C, 55.9; H, 4.00; Ru, 23.1; Si, 6.10. $C_{21}H_{18}O_3RuSi$ requires C, 56.3; H, 4.05; Ru, 22.7; Si, 6.25%). Mass spectrum: $M^+$, 448 (6); $[M - Me]^+$, 433 (0.1); $[M - CO]^+$, 420 (19); $[M - Me - CO]^+$, 405 (1); $[M - (CO)_2]^+$, 392 (15); $[M - Me - (CO)_2]^+$, 377 (1); $[M - (CO)_3]^+$, 364 (37); $[M - Me - (CO)_3]^+$, 349 (10).

*Interaction of* 1-*Chloro*-1,2,3,4,5-*pentaphenyl*-1-*silacyclopentadiene with Triruthenium Dodecacarbonyl.*—The silacyclopentadiene (0.25 g, 0.5 mmol) and the carbonyl (0.19 g, 0.3 mmol) were heated (12 h) under reflux in toluene. After removal of the toluene (20 °C, 0.1 mmHg), the residue was extracted with hot hexane, which on cooling gave yellow crystals of tricarbonyl(1-chloro-1,2,3,4,5-pentaphenyl-1-silacyclopentadiene)ruthenium, (10) (0.22 g, 64%), m.p. 196—201 °C (decomp.) (Found: C, 65.1; H, 3.75. $C_{37}H_{25}ClO_3RuSi$ requires C, 65.1; H, 3.70%). Mass spectrum: $M^+$, 682 (1); $[M - CO]^+$, 654 (3); $[M - (CO)_2]^+$, 626 (4.5); $[M - (CO)_3]^+$, 598 (10); $[C_4Ph_4SiPhCl]^+$, 496 (100).

Chromatography of the remaining hexane solution on Florisil gave an eluate which after recrystallisation from hexane gave tricarbonyl(1-hydroxy-1,2,3,4,5-pentaphenyl-1-silacyclopentadiene)ruthenium. Mass spectrum: $M^+$, 664 (0.6); $[M - CO]^+$, 636 (1.8); $[M - (CO)_2]^+$, 608 (2.5); $[M - (CO)_3]^+$, 580 (8); $[C_4Ph_4SiPh(OH)]^+$, 478 (100).

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